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(57) Abstract

The present invention relates to thermosetting powdered paints in various colors that contain bleeding pigments or colors which, when mixed together and subsequently applied with a particular two-coat cycle, allow to obtain uniform tints having a high hiding power that cannot be obtained with the thermosetting powdered paints used so far, which produce the phenomenon known as "salt-and-pepper" effect if their colors are different and they are mixed together.

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# THERMOSETTING POWDERED PAINTS AND TWO-COAT PAINTING PROCESS FOR THERMOSETTING POWDERED PAINTS

## Technical field

The present invention relates to thermosetting powdered paints, particularly for industrial applications, in various colors which, when mixed together and then applied with a particular two-coat cycle, allow to obtain uniform tints with high hiding power that cannot be obtained with the thermosetting powdered paints used so far, which produce the phenomenon known as "salt-and-pepper" effect if they are of different colors and mixed together.

## Background Art

Paints have been known and used since antiquity; they were initially produced by using oils, natural resins, colored earths and oxides, and have always been appreciated by mankind for their ability to protect and decorate everyday items.

The continuous search for new products and new application systems, as well as the need to reduce the pollutant content of liquid paints, which can be used only with solvents and more recently with small amounts of water, have led to the creation of thermoplastic powders that use high-molecular-weight resins.

However, these powders have demonstrated limits to their use.

A logical consequence of this has been the development of new low- and medium-molecular-weight polymers that can be catalyzed with hardeners by using heat to obtain thermosetting polymers which as such can no longer be

altered by the action of heat.

Such paints have been studied and tested in the mid-Sixties in the United States as technological (anticorrosion) epoxy powders and have enjoyed considerable development and diversification in Europe, especially in Germany and Italy, allowing them to meet the most disparate requirements.

Powdered paints currently used are, in order of decreasing utilization:

-- epoxy-polyesters

-- polyesters

-- epoxy paints

-- polyurethane paints

-- acrylic paints

The formulations of thermosetting powdered paints entail the use of raw materials (resins, catalysts, dilatants, deaerating agents, various additives, solid pigments, and fillers) that are specific for each system.

The production process includes the pre-mixing of all the components of the formulation and the extrusion of the mix, which allows to obtain perfect homogenization by virtue of the action of considerable shearing forces while heat is applied.

The extruded mass is immediately calendered to achieve the quickest possible cooling.

Subsequent milling reduces the material to a powder in which 99% of the grains measure less than 100 microns and no more than 5% of the grains measure less than 10 microns.

All these operations are checked constantly by using very modern state-of-the-art checking instruments.

Thermosetting powdered paints are applied in automatic or semiautomatic industrial plants that include the application booth, which fully recovers the product that has not adhered to the parts, the associated fluid bed for fluidifying the powder, and the system of movable or fixed electrostatic guns that dispense the powder through an electric field with the aid of low-pressure compressed air.

The objects to be coated are suspended from an aerial chain conveyor as they pass through the booth, where most of the dispensed electrically-charged powder adheres to the parts, which are sent to the curing oven.

The thicknesses that can be obtained, measured after curing, vary between approximately 40 and 80 microns.

As already tested in liquid paints, chemically different powders, too, are often mutually incompatible. Furthermore, if one mixes two pigmented powders of different colors, composed of compatible or incompatible resins, one obtains a powder that produces, when applied, a film of paint having a speckled appearance and an uneven color (salt-and-pepper effect). In summary, the advantages of the thermosetting powdered paints used so far are:

-- absence of volatile products (solvents)

-- quick, automatic and cheap execution of the painting cycle

-- coating with considerable thicknesses (40-80 microns) in a single coat

-- high mechanical strength and anti-corrosion resistance.

However, thermosetting powdered paints are not free from drawbacks.

Regardless of the chemical nature of the products, a mix of two or more powdered paints of different colors can produce, once applied, a film of powdered paint which has insufficient dilatation properties, loses sheen and always has a non-uniform color (salt-and-pepper effect). Accordingly it is not possible to obtain a coating of powdered paint that has a desired color or hue of a color and is uniform if one uses a mix of two or more conventional powdered paints of different colors.

Disclosure of the Invention

The aim of the present invention is to eliminate the drawbacks described above in known types of thermosetting powdered paint, providing a type of thermosetting powdered paint that allows to obtain paint coatings with a practically unlimited range of colors and hues of colors which are uniform and have great hiding power by using a limited number of basic colors.

This aim is achieved by a thermosetting powdered paint which comprises resins, at least one pigment or color, and a filler, wherein said pigment or color is a bleeding pigment or color.

Utilization of the property of a wide range of organic pigments or colors to bleed from a first coat of paint to a second overcoat allows to eliminate the drawbacks due to the "salt-and-pepper" effect and allows, by mixing a limited number of "basic" colors, to obtain an almost unlimited range of uniform colors with high covering power. This last concept is commonly referenced to as "tintometric system".

The term "bleeding", as used here, concerns the

characteristic of a paint product of releasing one or more substances that characterize the color and consequently modify the color itself. This characteristic of a paint product is usually considered a "defect" that leads to an "alteration" of the color of the final coat.

The Applicant has found that the "bleeding" characteristic of some colors or pigments, hereinafter referenced to as "bleeding colors or pigments" can be used to obtain a wide range of colors and hues of color which are uniform and have a high hiding power for coatings obtained by painting.

Advantageously, the paint according to the present invention furthermore comprises a metallic powder that gives a metallic appearance to the coating obtained by painting. The metallic powder is preferably constituted by aluminum powder.

Any pigment or color having bleeding properties and comprised within the classes of acid colors, naphthol combinations, basic colors, developers, direct colors, disperse colors, optical whitening agents, food-grade colors, developing colors, chrome colors, natural colors, oxidation bases, pigments, reactive colors, reducing agents, colors for solvents, sulfur colors, and vat colors, can be used as bleeding pigment or color.

Examples of preferred bleeding colors or pigments for providing the paints according to the present invention are chosen among the following materials of the Color Index, according to their generic names:

ZAPON GELB 156 CI SOLVENT YELLOW 21

| | |
|---|---|
| HANSA BRILLANT GELB 56x | CI PIGMENT YELLOW 74 |
| SOLVAPERM BLAU B | CI SOLVENT VIOLET 13 |
| SOLVAPERM ROSSO BB | CI SOLVENT RED 195 |
| SOLVAPERM ORANGE G | CI SOLVENT ORANGE 60 |
| THERMOPLAST GELB 104 | CI SOLVENT YELLOW 93 |
| SOLVAPERM GRUN G | CI SOLVENT GREEN 28 |
| SOLVAPERM ROTVIOLETT R | CI DISPERSE VIOLET 26 |
| SOLVAPERM GIALLO G | CI DISPERSE YELLOW 64 |
| MACROLEX BLAU RR | CI SOLVENT BLUE 97 |

Conveniently, the bleeding color or pigment is present in the paint according to the present invention in concentrations between 0.0001% and 50% by weight.

The range of bleeding color or pigment concentrations in the paint according to the present invention between 0.005 and 5% by weight is particularly preferred.

The resins used in paints according to the present invention are epoxy resins, polyester resins, polyurethane resins, or acrylic resins, individually or in combination with each other.

The thermosetting powdered paint according to the present invention preferably has an average powder particle size of 10 to 100 μm. Advantageously, 99% of the particles have dimensions of less than 100 μm and less than approximately 5% of the particles have dimensions of less than 10 μm.

Furthermore, the present invention provides a composition of thermosetting powdered paint that comprises a mixture of at least two thermosetting powdered paints which comprise a resin, a bleeding color or pigment, and a filler, wherein the at least two thermosetting powdered

paints contain bleeding colors or pigments of different colors.

The thermosetting powdered paints containing a bleeding color or pigment, according to the present invention, which are mixed to obtain the above mentioned composition or formulation may contain the same resins and fillers or different resins and/or fillers.

The powdered paints are mixed, in order to obtain the composition or formulation of paints according to the present invention, by using conventional powder mixing techniques, such as manual mixing, mixing with drum-turning machines, or mixing in fluid beds.

Manual mixing is performed (for example inside a bag or a bucket which are agitated manually) when small amounts are to be mixed by introducing, in a bag or bucket, the amounts of powdered paint, weighed to the desired quantities, and by mixing manually for approximately ten minutes, agitating the container to obtain a uniform mixture.

Mixing with drum-turning machines is performed in the case of large amounts by introducing in a drum the amounts of powdered paint to be mixed in the desired ratios and by making the drum revolve on appropriate devices (drum turning machines). Mixing lasts approximately ten minutes.

Mixing can also occur directly in a fluid bed after carefully checking the amounts of the individual components and after waiting, while the fluid bed is active, for approximately five minutes to obtain a uniform mixture.

In another embodiment of the present invention, a coating is provided which is based on thermosetting

powdered paints and comprises a first coat containing at least one thermosetting powdered paint, that contains a bleeding color or pigment according to the present invention, and a second coat of transparent or colored transparent thermosetting powdered paint.

Advantageously, the coating based on thermosetting powdered paints according to the present invention comprises a first coat, which contains a composition of paints according to the present invention constituted by a mix of thermosetting powdered paints that contain a bleeding color or pigment, and a second coat of transparent or colored transparent thermosetting powdered paint.

In another embodiment of the present invention, a two-coat painting process is provided which includes the following steps:

a) application of a first coat of thermosetting powdered paint, which comprises resin, fillers, and bleeding color or pigment, to the surface to be painted;

b) first curing in an oven to harden said first coat of paint;

c) application of a second coat of transparent thermosetting powdered paint on the first coat of hardened paint;

d) second curing in an oven to make the color or pigment of the first coat bleed, dissolving it uniformly inside the second coat while the second coat hardens.

The Applicant has found that the uniformity of the color and the hue of color of the final coating does not depend on the thickness of the two coats of paint applied.

The present invention furthermore provides a two-coat

painting process which is similar to the one described above except for the fact that the first coat contains a composition or formulation that includes at least two thermosetting powdered paints according to the present invention which comprise bleeding colors or pigments of different colors.

According to the present invention, a coat of priming powdered paint is applied which contains one or more bleeding colors or pigments with the optional addition of metallic pigments, pearly pigments, polyester glitter or fine metallic grains.

After performing appropriate curing, a second coat of transparent, colorless or colored powdered paint, with a degree of sheen that can vary between 5 and 110 gloss, measured with an angle of $60^{o}$, is applied over the first coat.

By virtue of the action of the heat, the colors or pigments bleed from the first coat in which they are contained and dissolve uniformly inside the second coat.

Accordingly, by applying a mix of two or more powdered paints that contain different bleeding colors or pigments a film is obtained that once overpainted has a new tint with a compact and uniform appearance and a high hiding power.

This phenomenon occurs independently of the chemical nature of the powdered paints and of the thicknesses of the applied coats.

It is therefore possible to superimpose coats of polyester, epoxy-polyester, epoxy, polyurethane or acrylic powdered paints according to the specific requirements, provided that they are further covered by the coat of

transparent or colored transparent paint.

By using a powdered paint that does not contain bleeding colors or pigments mixed with one or more powdered paints that contain bleeding colors or pigments (individual ones or different ones) it is possible to decrease the coloring force of the first coat.

The following examples are given only by way of example and are in no way meant to limit the invention.

Example 1

By mixing, in appropriate ratios, a powdered paint that contains a red color or pigment and a powdered paint that contains a blue color or pigment, a violet color is obtained that is more or less bluish or reddish according to the bleeding color or pigment that prevails in the mixture.

25 kg of a blue powdered paint (with tristimulus values X=10.35, Y=9.61, and Z=22) are mixed with 25 kg of a red powdered paint (tristimulus values X=18.65, Y=11.77, Z=13.87) in a fluid bed (0.75 m base x 0.5 m x 0.5 m height) with incoming air pressure at 1.5 atm. The mixing time is set to 5 minutes. At the end of the mixing, the powdered paint thus obtained is applied with an electrostatic gun on a metal part which is then placed in an oven set to 180°C and is cured for approximately 20 minutes.

Once cooled, the part is overpainted with a transparent powdered paint and returned to the oven for another 20 minutes at 180°C. At the end of the curing cycle, the part has a uniform violet color (tristimulus values X=9.16, Y=6.95, Z=15.57) which has a compact and

uniform appearance (maximum delta E between the different reading points is 0.5).

Readings were made with a spectrophotometer according to CIBLAB color space 1976, illuminate D 65 10° observer.

The tristimulus values (X Y Z) are calculated by means of the reflectances of the color being tested, correspond to the three primary colors, red (X), green (Y), and blue (Z), and are specific for each individual hue of color.

Example 2

By mixing, in appropriate ratios, a thermosetting powdered paint (which comprises a resin, a bleeding color or pigment, and a filler) and a thermosetting powdered paint (which comprises a resin, a filler and a metallic pigment) it is possible to decrease the coloring force of the "bleeding" paint.

25 kg of a blue powdered paint (with tristimulus values X=10.35, Y=9.61, and Z=22) are mixed with 25 kg of a pigment-free powdered paint in a fluid bed (0.75 m base x 0.5 m x 0.5 m height) with incoming air pressure at 1.5 atm.

The mixing time is set to 5 minutes. At the end of the mixing, the powdered paint thus obtained is applied with an electrostatic gun on a metal part which is then placed in an oven set to 180°C and is cured for approximately 20 minutes.

Once cooled, the part is overpainted with a transparent thermosetting powdered paint and returned to the oven for another 20 minutes at 180°C. At the end of the curing cycle, the part has a uniform light blue color (tristimulus values X=12.25, Y=12.25, Z=31.6) which has a

compact and uniform appearance (maximum delta E between the different reading points is 0.5).

Example 3

By mixing, in appropriate ratios, a thermosetting powdered paint (which comprises a resin, a bleeding color or pigment, and a filler), another thermosetting powdered paint (which comprises a resin, a bleeding color or pigment, and a filler), and a thermosetting powdered paint (which comprises a resin, a metallic pigment, and a filler), it is possible to obtain a tint that is not as intense as the "bleeding" tints used in the mix itself.

12.5 kg of a blue powdered paint (with tristimulus values X=10.35, Y=9.61, and Z=22) are mixed with 12.5 kg of a red powdered paint (with tristimulus values X=18.65, Y=11.77, and Z=13.87) and with 25 kg of a pigment-free powdered paint in a fluid bed (0.75 m base x 0.5 m x 0.5 m height) with incoming air pressure at 1.5 atm. The mixing time is set to 5 minutes. At the end of the mixing, the powdered paint thus obtained is applied with an electrostatic gun on a metal part which is then placed in an oven set to 180°C and is cured for approximately 20 minutes.

Once cooled, the part is overpainted with a transparent thermosetting powdered paint and returned to the oven for another 20 minutes at 180°C. At the end of the curing cycle, the part has a violet color (tristimulus values X=16, Y=15.2, Z=34.2) which has a compact and uniform appearance (maximum delta E between the different reading points is 0.5).

**Example 4**

By mixing, in appropriate ratios, a thermosetting powdered paint (which comprises a resin, a bleeding color or pigment, and a filler) and another thermosetting powdered paint (which comprises a resin, a non-bleeding pigment, and a filler) it is possible to obtain a tint having a uniform hue and appearance.

25 kg of a blue powdered paint that contains a bleeding pigment or color are mixed with 25 kg of a powdered paint that contains a non-bleeding color or pigment in a fluid bed (0.75 m base x 0.5 m x 0.5 m height) with incoming air pressure at 1.5 atm.

The mixing time is set to 5 minutes.

At the end of the mixing, the powdered paint thus obtained is applied with an electrostatic gun on a metal part which is then placed in an oven set to 180°C and is cured for approximately 20 minutes.

Once cooled, the part is overpainted with a transparent thermosetting powdered paint and returned to the oven for another 20 minutes at 180°C. At the end of the curing cycle, the part has a compact and uniform color.

These examples are also valid for thermosetting powdered paints having a surface finish other than the conventional smooth one. It is thus possible to mix orange-peel paints, textured paints, etcetera.

With the Arsonmix system it is also possible to extend the range of finishes with effects that are different from the source by adding other raw materials, maintaining the prerogatives of the primary Arsonmix system, which is the bleeding between a first and a second coats of

thermosetting powdered paint.

Thermosetting powdered paints according to the present invention, which contain a bleeding color or pigment, allow the user to obtain coatings with a practically unlimited range of colors or hues of color, chosen personally and according to one's individual preferences, by using a relatively small number of basic paints as starting materials. The compositions or formulations of thermosetting powdered paints that contain a mix of at least two thermosetting powdered paints according to the present invention can be prepared simply and directly by the user in the desired amounts (even very small ones).

Whereas in conventional coating and in the corresponding conventional two-coat painting process that uses thermosetting powdered paints the second transparent coat only acts as additional protection, in the coating and two-coat painting process according to the present invention the transparent coat has the task of making the color or pigment comprised in the first coat of paint "bleed", consequently dissolving into the second transparent coat. In this manner the thermosetting powdered paints according to the present invention allow to obtain a coating having a uniform color even when the first coat comprises a mix of paints of different colors, whereas in the conventional two-coat system if the first coat comprises a mix of thermosetting powdered paints with non-bleeding colors or pigment of different colors, the coating that is obtained does not have a uniform color, since the so-called "salt-and-pepper" effect is obtained.

The invention thus conceived is susceptible to

numerous modifications and variations, all of which are within the scope of the inventive concept.

## CLAIMS

1. Thermosetting powdered paint, comprising resins, at least one pigment or color, and a filler, characterized in that said pigment or color is a bleeding pigment or color.

2. Thermosetting powdered paint according to claim 1, characterized in that it furthermore comprises a metallic powder.

3. Paint according to claim 2, characterized in that said metallic powder is an aluminum powder.

4. Paint according to any one of the preceding claims, characterized in that it furthermore comprises multiple compounds chosen among the group constituted by metallic pigments, pearly pigments, polyester glitter, and fine metallic grains.

5. Paint according to claim 1, characterized in that said bleeding color or pigment is chosen from the group constituted by acid, azo, basic, developer, direct, disperse colors, food-grade optical whitening agents, developing, chrome, natural, basic oxidation, reactive, reducing, persolvent, sulfur, and vat bleeding colors, and bleeding pigments.

6. Paint according to claim 1, characterized in that said bleeding color or pigment is present at concentrations between 0.0001% and 50% by weight.

7. Paint according to claim 6, characterized in that said bleeding color or pigment is present at a concentration between 0.005% and 5% by weight.

8. Paint according to claim 1, characterized in that said resin is chosen from the group constituted by epoxy

resins, polyester resins, polyurethane resins, acrylic resins, either individually or combined with each other.

9. Paint according to claim 1, characterized in that its average powder particle size is between 10 and 100 μm.

10. Paint according to claim 9, characterized in that 99% of the powder particles have a size of less than 100 μm and less than 5% of the powder particles have a size of less than 10 μm.

11. Composition of thermosetting powdered paint, characterized in that it comprises a mix of at least two thermosetting powdered paints according to any one of the preceding claims, said at least two paints containing bleeding pigments or colors of different colors.

12. Coating based on thermosetting powdered paints, characterized in that it comprises a first coat of thermosetting powdered paint according to any one of claims 1 to 10, and a second coat of transparent or colored transparent thermosetting powdered paint.

13. Coating based on thermosetting powdered paints, characterized in that it comprises a first coat which is constituted by a composition of powdered paints according to claim 11 and a second coat of transparent or colored transparent thermosetting powdered paint.

14. Two-coat painting process which comprises the steps of:

a) applying to the surface to be painted a first coat of thermosetting powdered paint according to any one of claims 1 to 10;

b) first curing in an oven to harden said first coat of paint;

c) applying a second coat of transparent thermosetting powdered paint on the hardened first coat of paint;

d) second curing in an oven to cause the bleeding of the bleeding pigment or color contained in the first coat of paint in order to dissolve them uniformly inside the second coat of paint while said second coat hardens.

15. Two-coat painting process which comprises the steps of:

a) applying to the surface to be painted a composition of thermosetting powdered paints according to claim 11;

b) first curing in an oven to harden said first coat;

c) applying a second coat of transparent or colored transparent thermosetting powdered paint on the hardened first coat;

d) second curing in an oven to harden said second coat while the bleeding pigments or colors contained in the first coat bleed and dissolve uniformly in the second coat.

16. Process according to claim 14 or 15, characterized in that the step that includes the first curing in an oven is performed at a temperature between 120 and 240°C.

17. Process according to claim 14 or 15, characterized in that the step that includes the second curing in an oven is performed at a temperature in the range between 120 and 240°C.

18. Process according to claim 14 or 15, characterized in that the total thickness of the first and second coats after curing is in the range between 40 and 200 μm.

19. Process according to claim 14 or 15, characterized in that said transparent thermosetting powdered paint comprises resins chosen in the group constituted by epoxy

resins, polyester resins, polyurethane resins, acrylic resins, and mixes thereof.

20. Process according to claim 14 or 15, characterized in that said second coat of transparent thermosetting powdered paint has a sheen comprised between 5 and 110 gloss, measured at an angle of 60°.

21. Use of at least two thermosetting powdered paints according to any one of claims 1 to 10, wherein said at least two paints contain bleeding pigments or colors of different colors in order to obtain a coating in a color, or hue of color, that is different from the colors of the at least two thermosetting powdered paints, said coating having a uniform color.

# INTERNATIONAL SEARCH REPORT

Intern. nal Application No
PCT/EP 94/03411

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C09D5/03 B05D5/06 B05D7/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C09D B05D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category * | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| A | WO,A,91 18951 (COURTAULDS COATINGS) 12 December 1991<br>see abstract<br>see page 21, line 1 - line 22<br>see page 34, line 14 - line 23 | 1-4,6,8, 9,11,21 |
| A | EP,A,0 459 048 (COURTAULDS COATINGS) 4 December 1991<br>see abstract<br>see page 2, line 33 - line 47<br>see page 3, line 37 - line 58<br>see page 8 | 1,6,8,9, 11,21 |

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information on patent family members

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# INTERNATIONAL SEARCH REPORT

Intern. nal Application No
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

| Category * | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
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